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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention is obtained from the method and it which are low cost and manufacture an amide amine oxide compound with good hue and temporal stability of a scent, and relates to the good surface activity constituent, especially detergent composition of hue and the temporal stability of a scent.

[0002]

[Description of the Prior Art]An amide amine oxide compound has the useful stimulus to the skin as few surface-active agents like the amine oxides which are the allied substances.

It is a useful substance which is excellent in compatibility with sulfate system active agents, such as alkylbenzene sulfonates, and can be broadly used for detergents, such as detergent for kitchen and a shampoo, perfumery and cosmetics, cosmetics, etc. [ an amine oxide compound ].

As a manufacturing method of an amide amine oxide compound, the method of obtaining amine oxides, and the method to which hydrogen peroxide is made to react to an amide amine compound similarly are performed widely. However, in the solution of the amide amine oxide compound obtained by this method. Since the unreacted amide amine compound is contained so much, this has bad influence on the color tone of an amide amine oxide compound, a scent, a detergency, skin irritation, etc., and, for this reason, the detergent composition which used the amide amine oxide compound effectively actually is not yet developed.

[0003]On the other hand, in the case of the amine oxide compound which is allied substances, The problem is solved by adding hydroxyl content polybasic carboxylic acid, such as citrate, for the problem of the same unreacted amine as the above at the method given in JP,60-55060,B of composition of an amine oxide compound, i.e., the time, for example. When citrate was added in that system of reaction in accordance with the method of this gazette at the time of composition of an amide amine oxide compound, the rise was looked at by the oxide inversion rate of the amide amine compound as compared with the thing which does not add citrate, but. As a result of the abuse examination, change of coloring and a scent takes place simultaneously and, not only as for the amide amine oxide compound containing composition which was still excellent in stability to coloring and a smell but amide amine-oxide solution excellent in the stability of coloring and a scent, the generated amide amine oxide compound is not obtained. For this reason, the amide amine oxide compound was industrially used as the difficult substance of use.

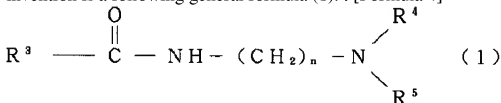
[0004]

[Problem(s) to be Solved by the Invention] This invention tends to provide the constituent containing the manufacturing method of a color tone and an amide amine oxide compound with the good temporal stability of a scent, and it. Especially this invention is low cost and tends to provide the method of manufacturing a color tone and an amide amine oxide compound with the good temporal stability of a scent and the constituent containing it, especially a detergent composition.

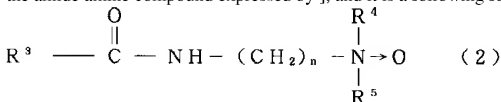
[0005]

[Means for Solving the Problem] This invention persons aim at obtaining a color tone and an amide amine oxide compound with a good scent, When oxidizing an amide amine compound and manufacturing an amide amine oxide compound wholeheartedly as a result of examination, it found out that an amide amine oxide compound without change of good hue stability and a scent was obtained by blending a specific organic-phosphonic-acid compound into this system of reaction. As a result of this invention persons' examining wholeheartedly the addition effect of said organic-phosphonic-acid compound, even if it did not add citrate, they succeeded in acquiring a citrate addition method of a statement, and an oxide appending rate more than equivalent to JP,60-55060,B. With phosphonic acid of a small quantity [ method / of blending specific organic phosphonic acid or its salt with amide amine-oxide solution of a high oxide inversion rate produced by adding citrate etc. ] or the amount of the salt used. It found out that a color tone and an amide amine oxide compound composition constituent with the good temporal stability of a scent were obtained. Furthermore, this invention persons checked that temporal stability was not spoiled even if it combines with other active agents an amide amine oxide compound water content solution obtained by this manufacturing method, and completed this invention.

[0006] A manufacturing method of an amide amine oxide compound with the good stability of this invention is a following general formula (1). : [Formula 4]

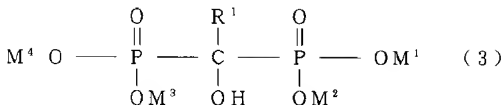


[However, in a formula (1), R<sup>3</sup> expresses the alkyl group or alkenyl group which is not replaced [ the hydroxyl substitution of 7-21, or ], and the number of carbon atoms R<sup>4</sup> and R<sup>5</sup>, Independently of \*\*, the number of carbon atoms expresses the alkyl group or alkenyl group of hydroxyl substitution or un-replacing of 1-5, and n expresses the integer of 1-5, respectively. Hydrogen peroxide is made to react to the amide amine compound expressed by ], and it is a following formula (2). : [Formula 5]



It faces manufacturing the amide amine oxide compound expressed by [however, R<sup>3</sup>, R<sup>4</sup>, R<sup>5</sup>, and n are the same as the above in a formula (2)], and is a following formula in said system of reaction (3). :

[Formula 6]



[However, among an upper type (3), as for  $\text{R}^1$ , a hydrogen atom or the number of carbon atoms expresses a low-grade alkyl group of 1-3, and  $\text{M}^1$ ,  $\text{M}^2$ ,  $\text{M}^3$ , and  $\text{M}^4$  express a hydrogen atom or an alkaline metal atom independently of others, respectively. Organic phosphonic acid expressed by ] or its salt is made to exist with 0.1 - 1.5-mol% of an addition to preparation molar quantity of an amide amine compound of said general formula (3). An amide amine oxide compound content surface activity constituent of this invention is obtained by said this invention method, and contains an amide amine oxide compound of said formula (2), and organic sulfonic acid of said formula (3) or its salt.

[0007]That is, in this invention method, a color tone and an amide amine oxide compound with a good scent can be obtained by low cost by blending specific organic phosphonic acid shown by a general formula (3), or its salt into the system of reaction. In a resultant mixture obtained by this invention method, It is useful especially as a detergent composition as an amide amine oxide compound content surface activity constituent whose color tone and scent of this constituent are good including organic phosphonic acid of a general formula (3), or its salt in an amide amine oxide compound of a general formula (2).

[0008]

[Embodiment of the Invention]In the manufacturing method of this invention, as an amide amine compound of the formula (1) used as a raw material, N,N-dimethylaminopropyloctylamide, N,N-dimethylaminopropyldodecylamide, N,N-dimethylaminopropyltetradecylamide, N,N-dimethylaminopropylhexadecylamide, N,N-dimethylaminopropyloctadecylamide, N,N-dimethylaminopropyl coconut oil alkylamide, N,N-dimethylaminopropyl hardening beef tallow alkylamide, N,N-dimethylaminopropyldodecylamide, N,N-bis(2-hydroxyethyl)propyldodecylamide, etc. are mentioned.

[0009]The natural oil fat whose above-mentioned amide amine compound is a condensate of fatty acid, fatty acid, and glycerin, Diamine like N and N dimethylamino propylamine can be made to be able to act on the refining fatty acid [ alcohol ester / fatty acid low-grade ] which passed through purification processes, such as molecular distillation and recrystallization, further, refining natural oil fat, refining fatty acid low-grade alcohol ester, etc., and it can obtain easily by an old acquaintance's method.

[0010]As the organic phosphonic acid of a general formula (3) used for this invention method, and its salt, hydroxyethanediphosphonic acid, hydroxypropane phosphonic acid, 1-hydroxybutane- 1, 1-JIHOSUHON, its salt, etc. can be used, for example.

[0011]Although water is generally used as a solvent of a reaction, in order to adjust the viscosity of amine-oxide solution, etc., hydrophilic solvents, such as methanol, ethanol, and 2-propanol, can be used together.

[0012]The amount of the organic phosphonic acid added in the system of reaction or the salt used of those is 0.1-1.5-mol % to the charge of an amide amine compound. Although it is possible to raise a reaction yield also with the addition below 0.1 mol %, the constituent containing an amide amine oxide compound and it with the satisfactory stability of hue and a scent cannot be obtained. When the addition is superfluously added exceeding 1.5-mol %, the constituent containing an amide amine oxide

compound and it with the satisfactory stability of hue and a scent is obtained, but. Working hours are further extended by disassembly of the hydrogen peroxide which organic phosphonic acid or the salt of those will exist superfluously in a constituent as carrier over, and the cost improvement effect becomes low, and exists superfluously.

[0013]As for the hydrogen peroxide used for oxidation of an amide amine compound, it is preferred to be used in the excessive amount beyond 110 mol % of the amount of theories to the quantity of the amide amine compound used. When hydrogen peroxide more superfluous than this is used, a reaction advances with high conversion, but in order to remove superfluous hydrogen peroxide, excessive cost will start. If the quantity of hydrogen peroxide is less than [ of the quantity of an amide amine compound / 100 mol % ], since an unreacted amide amine compound may remain and stimulativeness may be shown in a resultant, it is not desirable. Although there is no limitation in particular in the concentration of the hydrogen peroxide to be used, generally about 30% of hydrogen-peroxide-solution solution is easy to come to hand, and handling is also simple.

[0014]40 \*\* - 100 \*\* are suitable for the reaction temperature of an amide amine compound and hydrogen peroxide. They are 60 \*\* - 100 \*\* more desirably. Reaction temperature needs 6 hours or more by reaction conclusion at less than 40 \*\*, and is unsuitable to actual manufacture. Since the boiling point of the water used as the main solvent will be exceeded when reaction temperature exceeds 100 \*\*, the concentration adjustment of a product is needed and it is not desirable practically. The color protection by organic phosphonic acid or its salt also becomes insufficient.

[0015]The surface activity constituent containing the amide amine oxide compound and it which are obtained from the process of this invention is useful to liquid detergents, such as detergent for kitchen and a shampoo. When forming a detergent composition, an ampholytic surface active agent of a different kind, perfume, a coloring agent, a polymers thickener, a conditioning agent, an antiseptic, a hydrotrope agent, an ultraviolet ray absorbent, etc. can be arbitrarily blended with an anionic surface-active agent, a nonionic surfactant, and the compound of a formula (2).

[0016]When the amide amine oxide compound containing composition obtained by the manufacturing method of this invention is blended with oil insoluble to water at ordinary temperature like ethylene glycol distearate, The hue stability of the dispersion liquid of a pearly luster fats-and-oils crystallographic group is more stable as compared with the case where the constituent obtained with a different manufacturing method (for example, constituent which used citrate for the catalyst) from this invention is blended. As long as the ingredient blended with the amide amine oxide compound containing composition of this invention is used for a detergent, it may usually be what kind of thing, and there is no exceptional limitation in the kind.

[0017]As said anionic surface-active agent which can be blended with the detergent composition of this invention, alkyl (straight chain or branched chain) benzene sulfonate and alkyl (nature -- or) [ and ] A straight chain or branched chain ethersulfate, alpha olefin sulfonate, Alkyl sulfate, alkylphenol ethersulfate, Alkylamide ethersulfate, fatty acid soap, higher alcohol phosphate, Polyoxyethylene higher alcohol phosphate, polyoxyethylene higher-fatty-acid phosphate, N-acylamino acid salts, such as sulfonation higher fatty acid salt, a sulfonation higher-fatty-acid high-class alcohol ester salt, a higher alcohol sulfosuccinate salt, acyl methyltaurine, and N-long chain acyl glutamate, an acyl isethionic acid salt, etc. are mentioned.

[0018]As a nonionic surface active agent, fatty acid diethanolamide, fatty acid monoethanolamide, Polyoxyethylene alkyl ether, polyoxyethylene polyoxypropylene alkyl ether, polyoxyethylene fatty acid



asked for the inversion rate to the amide amine oxide of amide amine.

Column : Inertsil C-4, the Saiensu-Sha make, 4.6 mm x phi150-mm eluate: -- mixed liquor measured wavelength [ of 0.025M NaH<sub>2</sub>PO<sub>4</sub>(pH:7):CH<sub>3</sub>CN=65:35 ]: -- 210-mm flow: -- 1 ml/min temperature: -- 40 \*\* hue evaluation: -- O colorlessness. O Evaluation of light yellow, \*\* light yellow - yellow, and x orange and a xx brown scent : unpleasant smells, such as amine and other pungent smells, become weak in order of xx->x->\*\*->O->O.

[0025]When chelated additive agents other than hydroxyethanediphosphonic acid were added and it reacted, it has checked that reaction conversion rose, as Table 1 showed, but the temporal stability of the initial color phase and the scent was poor. Also when the additive of a phosphoric acid system or a phosphorous acid system was used, the effect like hydroxyethanediphosphonic acid was not seen. It was checked that especially an aging preventive effect is an effect peculiar to hydroxyethane JIHON acid.

[0026]400 g (N,N-dimethylpropylauric acid which makes a subject Lauring by whom the average molecular weight 298 was got from amine value) of example 2 lauryl amide propyldimethylamine, The hydrogen peroxide solution 159.4g (amine 105 mol % for amide) was dropped at the aqueous suspension containing the water 843g over 1 hour 30%, having added the hydroxyethanediphosphonic acid 2.30g (amine 0.5 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, this was cooled on the room temperature, and the 30% solution of the amide amine oxide compound was obtained. Thus, reaction conversion was 99.7% as a result of analyzing the acquired output. Although abuse processing of this output was carried out for three months in a 50 \*\* thermostat, change of hue and a scent was not accepted. The combination experiment which mentions this output later was presented.

[0027]To the aqueous suspension containing 314 g (coconut-fatty-acid amide amine from which the average molecular weight 322.5 was obtained from amine value) of example 3 palm-oil amide propyldimethylamine, and the water 374.9g. The hydrogen peroxide solution 110.7g (amine 105 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 1.60g (amine 0.5 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, and amide amine-oxide 40% solution was obtained. Thus, reaction conversion was 99.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change of hue and a scent was not accepted.

[0028]To the aqueous suspension containing 200.0 g (myristic acid amide amine from which the average molecular weight 337.81 was obtained from amine value) of example 4 myristic-acid amide dimethyl propylamine, and the water 772.5g. The hydrogen peroxide solution 73.8g (amine 110 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 1.02g (amine 0.5 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, and amide amine-oxide 20% solution was obtained. Thus, reaction conversion was 98.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change of a scent and hue was not accepted.

[0029]To the aqueous suspension containing 200.00 g (formula mass 228.3) of example 5 refining capric acid amide propyldimethylamine, and the water 401.1g. The hydrogen peroxide solution 109.2g (amine 110 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 1.84g (amine 1.0 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. Temperature was raised to 90 \*\* after the end of dropping, it stirred for further 4 hours,

and amide amine-oxide 30% solution was obtained. Thus, reaction conversion was 99.7% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change of a scent and hue was not accepted.

[0030]To the aqueous suspension containing 98.50 g (formula mass 384.6) of example 6 refining 12 hydroxystearic acid AMIDOJI methylpropyl amine, and the water 894.2g. The hydrogen peroxide solution 31.93g (amine 110 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 1.32g (amine 1.5 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, and amide amine-oxide 10% solution was obtained. Thus, reaction conversion was 97.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change of a scent and hue was not accepted.

[0031]To the aqueous suspension containing 200.0 g (castor oil fatty-acid-amide amine from which the average molecular weight 393.64 was obtained from amine value) of example 7 castor-oil fatty acid AMIDOJI methylpropyl amine, and the water 474.7g. The hydrogen peroxide solution 66.5g (amine 105 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 1.92g (amine 1.0 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, and amide amine-oxide 30% solution was obtained. Thus, reaction conversion was 98.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change of a scent and hue was not accepted.

[0032]To the aqueous suspension containing 200.0 g (formula mass 298.5) of example 8 refining lauric acid amide diethylethylamine, and the water 420.4g. The hydrogen peroxide solution 79.8g (amine 105 mol % for amide) was dropped over 1 hour 30%, having added the hydroxyethanediphosphonic acid 2.30g (amine 1.0 mol % for amide) 60%, and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, it stirred for further 4 hours, and amide amine-oxide 30% solution was obtained. Thus, reaction conversion was 98.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change was not observed in hue and a scent.

[0033]To the aqueous suspension containing 400 g (formula mass 284.4) of example 9 refining lauryl amide propyldimethylamine, and the water 828.4g. The hydrogen peroxide solution 175.4g (amine 110 mol % for amide) was dropped over 1 hour 30%, having added the hydroxypropane diphosphonic acid 4.64g (amine 1.5 mol % for amide), and keeping temperature at 60 \*\* - 70 \*\*. After the end of dropping, temperature was raised to 90 \*\*, and it stirred for further 4 hours, and cooled to the room temperature, and amide amine-oxide 30% solution was obtained. Thus, reaction conversion was 97.5% as a result of analyzing the acquired output. Although this output was abused for three months in a 50 \*\* thermostat, change was not observed in hue and a scent.

[0034]The kitchen detergent of the presentation of a statement was prepared to Table 2 using the amide amine oxide compound (AAO) prepared in each of the comparative examples 7-12, the example 10 aforementioned comparative examples 1-6, and Example 2, this was saved a 40 \*\* homiothermal in a plane one, and change of hue and a scent was evaluated three months afterward. The result is shown in Table 2.

[0035]

[Table 2]

実施例No. 項目	比較例 7	比較例 8	比較例 9	比較例10	比較例11	比較例12	実施例10
使用 A A O 製造	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	実施例 2
S L E S 7 0 % 水溶液	21.4	21.4	21.4	21.4	21.4	21.4	21.4
A A O 3 0 % 水溶液	40	40	40	40	40	40	40
Cocamide D E A	3	3	3	3	3	3	3
エタノール	5	5	5	5	5	5	5
D P G	5	5	5	5	5	5	5
精製水	25.6	25.6	25.6	25.6	25.6	25.6	25.6
初期色相	×	×	○	○	○	◎	◎
初期香り	×	×	△	○	○	○	◎
40℃ 3 ヶ月後の色相	×	×	△	△	△	○	◎
40℃ 3 ヶ月後の香り	×	×	△	△	△	○	◎

[0036]\*\* SLES of Table 2: Lauryl ether sulfate 70% solution AAO: It is the same as evaluation of the lauryl AMIDOPUROPURU dimethylamine oxide DPG:dipropylene glycol hue and the scent which were prepared in each of the comparative examples 1-6 and Example 2, and \*\* of Table 1 to a statement.

[0037]The stability of the hue and the scent of a detergent composition inherits the stability of the used amide amine oxide compound as it is from the result of Table 2. Namely, the constituent (example 10) which uses the amide amine oxide which hydroxyethanediphosphonic acid was made to live together in the system of reaction, and the amide amine oxide was oxidized, and was compounded. It was checked that the amide amine oxide compound which is remarkably excellent as compared with what is depended on other manufacturing conditions, therefore was manufactured by this invention method is useful as an ingredient of a detergent composition.

[0038]The amide amine oxide compound prepared in example 11 Example 2 was used, and the shampoo of the following presentation was prepared. Although this was saved at a 40 °C homoiothermal machine and evaluated three months afterward, scent change and coloring were not accepted.

Cocoyl imidazolium betaine liquid (30% solution \*3). 33.33% lauryl ethereal sulfate Na (25% solution). 10.00% amide amine oxide 41.67% concentrated glycerin 3.00% cation-ized cellulose 0.10% succinyl carboxymethyl chitosan liquid (2% solution \*4) 0.50% methylparaben 0.20% pH adjuster Quantity purified water set to pH=7 Residue [\*\*]

\*3: Kawaken Fine Chemicals Co., Ltd. make SOFUTAZORIN CL\*4: Kawaken Fine Chemicals Co., Ltd. make Quito Aqua[0039]The amide amine oxide prepared in example 12 Example 2 was used, and the pearl gloss shampoo of the following presentation was prepared. Although this was saved at a 40 °C homoiothermal machine and evaluated three months afterward, change was not observed in scent change and pearly luster.

Lauryl ethereal sulfate Na (25% solution). 10.00% coconut-fatty-acid diethanolamide . 3.00% lauric acid amide propylbetaine liquid (30% solution \*5) 20.00% amide amine oxide (30% solution) 20.00%



succinyl carboxymethyl chitosan liquid (2% solution) 1.50% ethylene glycol distearate . 2.70% ethylene glycol monostearate 0.30% pH adjuster Quantity purified water set to pH=6.5 Residue [\*\*]

\*5: Kawaken Fine Chemicals Co., Ltd. make SOFUTAZORIN LPB[0040]The amide amine oxide compound prepared in example 13 Example 2 was used, and the concentration mold plate place detergent of the following presentation was prepared. Although this was saved at a 40 \*\* homoiothermal machine and evaluated three months afterward, change of a scent and change of hue were not accepted. Straight chain lauryl benzenesulfonic acid Na (50%). 30.0% amide amine oxide (30%) 50.0% dipropylene glycol 8.0% methylparaben 0.2% ethanol 5.0% atelocollagen (1% solution) 1.0% pH adjuster Quantity purified water set to pH=7.5 Residue[0041]

[Effect of the Invention]According to this invention method, amide amine oxide solution with good hue and smell can be obtained, and the detergent which has the outstanding temporal stability can be further obtained using the amide amine-oxide containing composition obtained by this method.

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[Translation done.]



said general formula (1).

[Claim 2]An amide amine oxide compound content surface activity constituent which is obtained by a method according to claim 1, and contains an amide amine oxide compound of said formula (2), and organic sulfonic acid of said formula (3) or its salt.

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[Translation done.]